

OPERATING INSTRUCTIONS FOR INTERNATIONAL SCIENTIFIC INSTRUMENTS MODEL  
DS-130 SCANNING ELECTRON MICROSCOPE AND TRACOR NORTHERN  
SERIES II X-RAY ANALYZER  
FOR EVALUATION OF YMP GEOLOGIC MATERIALS

Effective Date 5/31/89

Daid Dorton for Roland Hagan

Roland Hagan  
Preparer

5/30/89  
Date

Schön Levy

Schön Levy  
Technical Reviewer

5/26/89  
Date

Henry Paul Nunes

H. P. Nunes  
Quality Assurance Project Leader

5/30/89  
Date

R. J. Herbst

R. J. Herbst  
Technical Project Officer

5/30/89  
Date

8912190190 891211  
PDR WASTE  
WM-11  
PDC

**OPERATING INSTRUCTIONS FOR INTERNATIONAL SCIENTIFIC INSTRUMENTS MODEL  
DS-130 SCANNING ELECTRON MICROSCOPE AND TRACOR NORTHERN  
SERIES II X-RAY ANALYZER  
FOR EVALUATION OF YMP GEOLOGIC MATERIALS**

**1.0 PURPOSE**

This procedure provides instructions for the operation of the International Scientific Instruments (ISI) Model DS-130 scanning electron microscope (SEM) and Tracor Northern (TN) Series II x-ray analyzer providing energy-dispersive x-ray fluorescence spectrometry (EDXRFS) and an image analysis system (IAS). The SEM system is used to obtain high-magnification images of geologic materials, to determine the identities and distributions of elements in samples, and to characterize particulate samples.

**2.0 SCOPE**

This procedure applies to geologic specimens and other materials documented as Yucca Mountain Project (YMP) samples that are studied using the SEM.

**3.0 DEFINITION**

The machine custodian is in charge of the SEM and is responsible for its maintenance.

**4.0 REFERENCES**

- 4.1 TWS-QAS-QP-02.2: Procedure for Personnel Training
- 4.2 TWS-QAS-QP-3.1: LANL YMP Computer Software Control
- 4.3 TWS-QAS-QP-03.5: Procedure for Documenting Scientific Investigations
- 4.4 TWS-QAS-QP-12.1: NNWSI Instrument Calibrations
- 4.5 TWS-QAS-QP-13.1: Handling, Storage and Shipping Procedure
- 4.6 TWS-ESS-DP-06: Operating Instructions for DV-502 Vacuum Evaporator for Carbon Coating Samples
- 4.7 TWS-ESS-DP-50: Operating Procedure for Gold Coating Samples Using the Technics Hummer II Sputtering System
- 4.8 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies

## 5.0 APPLICABLE DOCUMENTS

- 5.1 International Scientific Instruments, Milpitas, CA
  - 5.1.1 Instruction Manual for ISI's Model DS-130 SEM
- 5.2 Akashi Seisakusho, Ltd., Japan
  - 5.2.1 Instruction Manual, SEM provided with a LaB6 [sic] ion source
- 5.3 Tracor Northern, Middleton, WI
  - 5.3.1 Series II X-Ray Analyzer Operator's Manual
  - 5.3.2 Software programs

	<u>Program Title</u>	<u>Location on disk</u>
5.3.2.1	1-1E/80 Prog Search	File 1
5.3.2.2	FIC-3J/80 File Copy	File 1627
5.3.2.3	IDENT-6F/80	File 3668
5.3.2.4	INIT 1K/80 EAPROM M	File 2668
5.3.2.5	BENCE-ALBEE-8G/80	File 161
5.3.2.6	ATABLE-1D/37 BA COEF	File 1909
5.3.2.7	CBEAM-1C/80 INST BE	File 2533
5.3.2.8	DL-1D/90 MICROQ DOWN	File 188
5.3.2.9	MICROQ-3J/80	File 3993
5.3.2.10	TREE-1D/80 MICROQ TR	File 317
5.3.2.11	QFILE 2I/80	File 821
5.3.2.12	DL2-1B/80 SSQ DOWN L	File 1506
5.3.2.13	SQ-2B/80	File 313
5.3.2.14	SQC-2B/80	File 2520
5.3.2.15	TREESQ-2B/80 SQ.TREE	File 121
5.3.2.16	SSQ-3L/80 SEMI-QUANT	File 2553
5.3.2.17	PRZ-1G/80 PROZA CORR	File 2362
5.3.2.18	ZAF-12L/80 ZAF CORR.	File 2846
5.3.2.19	VISTA-1C/80.MAIN	File 3057
5.3.2.20	TN5525 1D/80	File 2901
5.3.2.21	VHT-1A V HIST CMD	File 2660
5.3.2.22	PRC-6L/80.MAIN	File 2339
5.3.2.23	PRP-2C/80.MAIN	File 2352
5.3.2.24	VISTATR-1D/80 CMDTRE	File 3458
5.3.2.25	CALC-1C/80 1/30/87	File 2867

## 6.0 RESPONSIBILITIES

The machine custodian maintains the SEM system, instructs investigators in the safe use of the system, and certifies investigators for this procedure. The machine custodian is also responsible for maintenance of software and electronically-stored data. Materials are selected for study by a YMP principal investigator (PI). The PI has the responsibility to assure implementation of this procedure and may delegate performance of the procedure to any individual who is certified for the procedure. The PI also determines the acceptability of data produced by implementation of this procedure and is responsible for the proper storage and disposition of hard-copy data records.

## **7.0 PROCEDURE**

### **7.1 System Description**

The SEM (purchase date September 1980) is fully described in the ISI Model DS-130 operator's manual. The electron source was modified (installed March 1987) to be a dual source for tungsten or lanthanum boride and is described in the Source Operator's Manual.

The Analyzer (EDXRFS and IAS) is a Tracor Northern Series II x-ray analyzer (purchase date August 1987). This equipment is described in the TN operator's manual. The accompanying software supplied by TN (listed in section 5.2.2) has not been modified and is commercial software as defined by TWS-QAS-QP-3.1.

### **7.2 Sample Preparation**

The sample to be examined in the SEM must be in a form that can be inserted into one of the stage mounts stored next to the SEM. Before being placed in a stage mount, a sample must be given an electrically conductive coating following instruction in the procedures for SEM sample coating (TWS-ESS-DP-06 and TWS-ESS-DP-50). The PI determines what type of sample mount and conductive coating is appropriate.

### **7.3 Sample Identification**

SEM sample stubs or thin sections are marked with a YMP control number. If it is not practical to place the entire YMP control number on the sample mount, an abbreviated number may be placed on the mount. The abbreviated number, accompanied by the complete YMP control number, is recorded in the SEM logbook as part of the work description.

### **7.4 SEM Operation**

Before beginning an SEM examination, the investigator consults the system operator to verify that the SEM system is working properly. Initiation of a work entry in the SEM logbook documents the verification. The SEM is set up and operated according to the instructions contained in the ISI operating manuals and supplemental instructions posted on the SEM console. The manuals are stored in the laboratory near the instrument.

### **7.5 TN System Operation**

The TN system is set up and operated according to the instructions contained in the ISI operating manuals. These manuals are stored in the laboratory near the instrument.

### **7.6 Data Acquisition**

SEM photomicrographs are produced following the instructions in the operator's manuals. Each photomicrograph is labelled with the original sample number. At the analyst's option, the date and additional information may also be recorded on the photomicrograph.

The TN system collects data that are displayed on a monitor and may, at the operator's discretion, be recorded on floppy or hard disk and/or printed. The monitor display may be photographed. The operator decides what data records are needed. Electronic data files are

labelled with the original sample identification or with an alphanumeric designation keyed to the original sample identification in the SEM logbook. Instant photographs of the monitor display are labelled with the original sample number. Photographs taken on slide or print film may be labelled directly on the monitor display or logged in the analyst's laboratory notebook.

#### 7.7 Logbook and Notebook Entries

An operator using the SEM enters the date of use and identification of samples studied in the SEM logbook. Additional information, as appropriate, may include the identification of data files generated. At the operator's discretion, descriptions of work performed or observations are recorded in the operator's laboratory notebook, along with the date and identification of samples.

#### 7.8 Environmental Conditions

Normal interior building temperature and humidity are acceptable for the operation of the SEM system. Compressed air in the range of 50 to 80 psi is supplied by the building utilities. Cooling water for the SEM vacuum pump is supplied by a chilled water system maintained in the range of 40 to 60°F. All of these conditions are remotely monitored by Engineering Group ENG-5, and an alarm is automatically set off if an unacceptable condition is detected.

#### 7.9 Acceptance/Rejection Criteria

The investigator is responsible for acceptance or rejection of any photomicrograph or TN data output. Photomicrograph acceptability is subjective; no documentation is required for rejection of photomicrographs. The only general criterion for rejection of TN data output is instrument instability, which inserts readily-detectable lines of garbled data into maps displayed on the monitor. Beyond this, each investigator determine her/his own criteria for acceptance and rejection, which are documented in the laboratory notebook.

### 8.0 QUALITY ASSURANCE

#### 8.1 Records

YMP notebooks, logbooks, photomicrographs, TN data output, and other records are controlled in accordance with the procedure for documenting scientific investigations (TWS-QAS-QP-03.5) and the procedure for the LANL group resident file (TWS-QAS-QP-17.1). Photomicrographs and TN data output are labelled as specified in Section 7.7. Data output are retained by the PI as long as required. Any data output that qualifies as a one-of-a-kind record is controlled as such.

#### 8.2 Personnel Training

Training consists of reading this procedure and hands-on operation of the equipment under the supervision of the machine custodian or other qualified user. The machine custodian's signature will certify personnel for the use of this equipment. Training is documented in accordance with the procedure for personnel training (TWS-QAS-QP-02.2).

### **8.3 Calibration**

- 8.3.1** The magnification of the sample image on the SEM screen and photomicrographs is checked annually by the machine custodian by use of an NBS SEM magnification standard. Results are documented in the SEM logbook. The results, date, and name of the person performing the check are recorded in the SEM logbook.
- 8.3.2** The TN EDXRFS is checked monthly by following the energy calibration instructions in the TN operator's manual. The results, date, and the name of the operator performing the check are recorded in the SEM logbook.
- 8.3.3** Evidence of calibration is documented in accordance with the instrument calibration procedure (TWS-QAS-QP-12.1).

### **8.4 Documentation of Procedural Deviations**

Special research needs may require deviations from this procedure. The operator documents any deviations and the reasons that they were necessary in the SEM logbook or the operator's notebook, as required by the procedure for documenting scientific investigations (TWS-QAS-QP-03.5).

### **8.5 Sample Handling, Shipping, and Storage**

Handling, shipping, and storage of scientific samples is conducted according to TWS-QAP-QP-13.1 (handling, shipping, and storage procedure) and TWS-ESS-DP-101 (sample identification and control for mineralogy-petrology studies). Sample identification for SEM materials is described in Section 7.3.

### **8.6 Limitations**

Quantitative elemental analysis requires a precise set of conditions that are very difficult to meet in the instrument. All analysis work is to be considered semi-quantitative and is to be presented as such in any report.